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# The first phosphoramide–mercury(II) complex with a $Cl_2Hg–OP[N(C)(C)]_3$ segment

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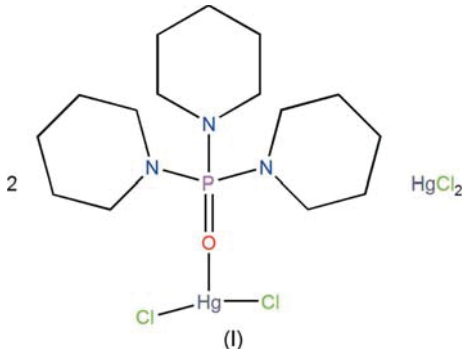
Mercury(II) exhibits a strong preference for linear coordination which has been attributed to relativistic effects splitting the 6*p* orbitals and promoting *sp* hybridization. If the two ligands attached to the mercury(II) ion are weak donors, the metal ion can act as a good Lewis acid and expand its coordination number. Moreover, mercury has a special affinity for softer bases, such as S and N atoms, and has much less affinity for hard bases, such as those including an O atom. The asymmetric unit of dichlorido[tris(piperidin-1-yl)phosphane oxide-κ*O*]mercury(II)–dichloridomercury(II) (2/1), $[HgCl_2\{(C_5H_{10}N)_3PO\}]_2\cdot[HgCl_2]$, is composed of one $HgCl_2\{(C_5H_{10}N)_3PO\}$ complex and one half of a discrete $HgCl_2$ entity located on an inversion centre. The coordination environment around the $Hg^{II}$ centre in the complex component is a distorted T-shape. Bond-valence-sum calculations confirm the three-coordination mode of the $Hg^{II}$ atom of the complex molecule. The noncovalent nature of the Hg···Cl and Hg···O interactions in the structure are discussed.

## 1. Introduction

Mercuric coordination compounds including ligands with different donor atoms (typically, sulfur, nitrogen and oxygen) have been investigated (Lippard, 1990). The +2 oxidation state is the most common for Hg and is also the main oxidation state of mercury in nature. The coordination chemistry of mercury(II) differs from most other transition metals due to its $d^{10}$ configuration and large size (Sahebalzamani *et al.*, 2010).




(I)

Mercury(II) exhibits a strong preference for linear coordination (Canty & Marker, 1976) which has been attributed to relativistic effects splitting the 6*p* orbitals and promoting *sp* hybridization (Thomas & Gaillard, 2015). It should be noted that if the two ligands attached to the mercury(II) ion are weak donors, *e.g.* chloride in $HgCl_2$, the metal ion can act as a good Lewis acid and expand its coordination number (Fisher

& Drago, 1975). Moreover, mercury has a special affinity for softer bases, such as S and N atoms, and has much less affinity for hard bases, such as those including an O atom (Meyer & Nockemann, 2003).

In this paper, we describe the synthesis and structural characterization of a new three-coordinated mercury(II) complex with a tris(piperidin-1-yl)phosphane oxide *O*-donor ligand. A search of the Cambridge Structural Database (CSD, Version 5.36, with updates to November 2014; Groom & Allen, 2014) revealed that: (i) the structure of (I) is the first example of a three-coordinated $Hg^{II}$ complex with a $Cl_2Hg—O{=}P[N(C)(C)]_3$ segment; (ii) there are only two phosphoric triamide $Hg^{II}$ structures present, namely bis(dimesylamide-$\kappa N$)(hexamethylphosphoramide-$\kappa O$)mercury(II) (CSD refcode JUWNOE; Blaschette *et al.*, 1993) and bis($\mu_3$-hexamethylphosphoramide)tris($\mu_2$-perfluoro-*o*-phenylene)trimercury(II) (CAMFEC; Tikhonova *et al.*, 2002), the preparation of both of which used the commercial material $[(CH_3)_2N]_3PO$; (iii) the $(C_5H_{10}N)_3PO$ ligand appears in only two complex structures, namely tris(isothiocyanato)tris(tripiperidinylphosphine oxide)-praseodymium(III) (MALMAO; da Silva *et al.*, 2005) and bis(nitrato-$\kappa^2O,O'$)dioxidobis(tripiperidinylphosphine oxide-$\kappa O$)uranium (LOPVUH; de Aquino *et al.*, 2000).

## 2. Experimental

### 2.1. Synthesis and crystallization

**2.1.1. Synthesis of tris(piperidin-1-yl)phosphane oxide, $(C_5H_{10}N)_3PO$.** The preparation of tris(piperidin-1-yl)phosphane oxide was reported previously as the by-product of the synthesis of $[(C_5H_{10}N)_4P]Br$ (Schiemenz *et al.*, 2001). The synthesis of $(C_5H_{10}N)_3PO$ in the present case was performed in a different way. A dry acetonitrile solution of piperidine (1.2 *M*, 10 ml) was added dropwise to a solution of $P(O)Cl_3$ (2 mmol) in the same solvent (30 ml) at 273 K. After stirring for 4 h, the precipitated amine hydrochloride salt ($C_5H_{10}NH{\cdot}HCl$) was filtered off and the acetonitrile solution of tris(piperidin-1-yl)phosphane oxide was used in the subsequent preparation of the title compound.

**2.1.2. Synthesis of $[HgCl_2\{(C_5H_{10}N)_3PO\}]_2{\cdot}[HgCl_2]$, (I).** A solution of $HgCl_2$ (1 mmol) in methanol (10 ml) was added dropwise to the acetonitrile solution of $(C_5H_{10}N)_3PO$ (2 mmol, 50 ml) and the resulting solution stirred for 48 h under reflux. After cooling to room temperature, colourless block-shaped crystals of (I) were obtained within a few days.

### 2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were included in calculated positions and treated as riding atoms, with C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. Attempts to model the disorder of the piperidine ring containing atom N2 were unsuccessful. Instead rigid-bond and similarity restraints were applied to atoms C6, C7, C8, C9 and C10.

**Table 1**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[HgCl_2(C_{15}H_{30}N_3OP)]{\cdot}[HgCl_2]$ |
| $M_r$ | 1413.25 |
| Crystal system, space group | Monoclinic, $P2_1/n$ |
| Temperature (K) | 300 |
| $a, b, c$ (Å) | 11.1182 (15), 10.5995 (8), 19.338 (3) |
| $\beta$ (°) | 96.337 (11) |
| $V$ (Å$^3$) | 2265.0 (5) |
| $Z$ | 2 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 10.60 |
| Crystal size (mm) | 0.23 × 0.16 × 0.10 |
| | |
| Data collection | |
| Diffractometer | Stoe IPDS 2 |
| Absorption correction | Integration (*X-SHAPE*; Stoe & Cie, 2002) |
| $T_{min}$, $T_{max}$ | 0.066, 0.133 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 27440, 4041, 3336 |
| $R_{int}$ | 0.061 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.599 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.036, 0.074, 1.05 |
| No. of reflections | 4041 |
| No. of parameters | 223 |
| No. of restraints | 31 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.66, −0.79 |

Computer programs: *X-AREA* (Stoe & Cie, 2009), *X-RED32* (Stoe & Cie, 2009), *SUPERFLIP* (Palatinus & Chapuis, 2007), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 1999) and *ORTEP-3 for Windows* (Farrugia, 2012).

## 3. Results and discussion

The asymmetric unit of (I) is composed of one $HgCl_2$-$\{(C_5H_{10}N)_3PO\}$ complex and one half of a $HgCl_2$ component (Fig. 1). In the discrete $HgCl_2$ component, the Hg2 atom is located on an inversion centre and hence shows an ideal linear coordination environment.

The Hg1—Cl bond lengths in the mercury(II)–phosphoric triamide complex of (I) are consistent with those reported for other complexes of $Hg^{II}$ chloride (Li *et al.*, 2012; Kubo *et al.*, 2000) and are longer than the two equal Hg—Cl bond lengths in the discrete $HgCl_2$ entity (Table 2). The Hg1—O1 bond length is comparable to similar bonds in analogous structures (Yang *et al.*, 2010).

In the $HgCl_2\{(C_5H_{10}N)_3PO\}$ complex, Cl1—Hg1—Cl2 is distorted by about 16° from a linear bonding arrangement due to the presence of the coordinated phosphoric triamide molecule. The coordinated O atom forms Cl1—Hg1—O1 and Cl2—Hg1—O1 angles that deviate from 90° towards higher angles, by about 16° and less than 1°, respectively (Table 2).

Khavasi & Azhdari Tehrani (2013) introduced the simple equation $\tau_3 = |[(\alpha + \beta + \gamma)/360] - |(\alpha - 120)/60||$ for the calculation of geometry index $\tau_3$ for three-coordinated compounds. In this equation, $\alpha$ is the largest angle in the three-coordinated compound and the values of $\tau_3$ range from 1.00 for a perfect trigonal planar geometry to zero for a perfect T-shaped geometry. The $\tau_3$ index for the three-coordinated

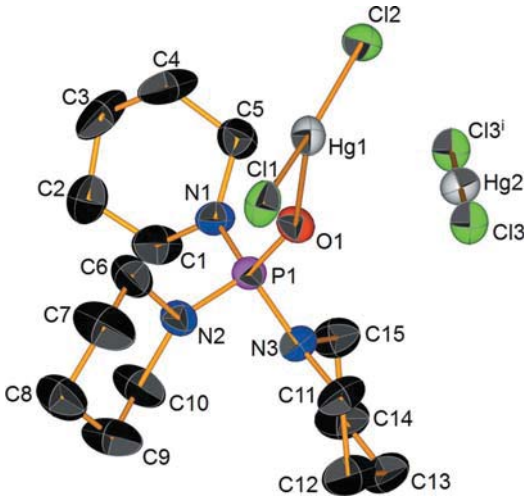



**Figure 1**
Displacement ellipsoid plot (30% probability level) for the [$HgCl_2$-{$(C_5H_{10}N)_3PO$}] and $HgCl_2$ components in (I), showing the atom-labelling scheme. H atoms have been omitted for clarity. [Symmetry code: (i) $-x, -y + 2, -z + 2$.]

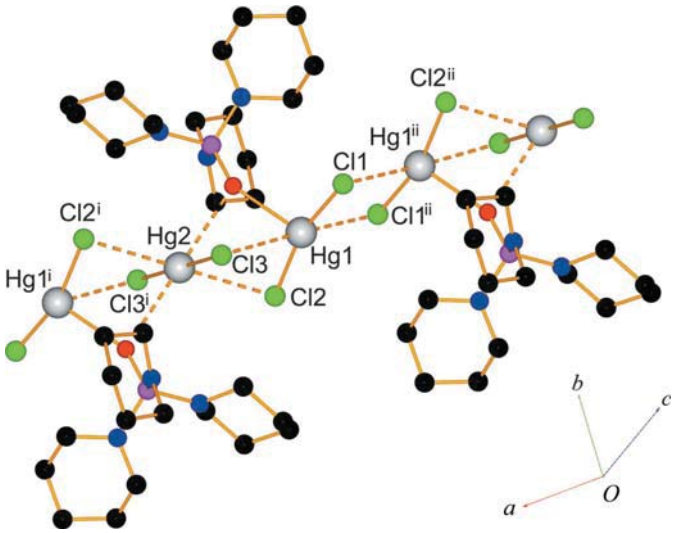



**Figure 2**
A view of linear arrangement of components parallel to [100] built from Hg···Cl and Hg···O interactions (dashed lines). H atoms have been omitted for clarity. [Symmetry codes: (i) $-x, -y + 2, -z + 2$; (ii) $-x + 1, -y + 2, -z + 2$.]

Hg1 complex of (I) is 0.27, which implies a distorted T-shape geometry (Tiekink, 1987).

Using a van der Waals radius of 1.70 Å for mercury, 1.75 Å for chlorine and 1.50 Å for oxygen (Batsanov, 2001), four different noncovalent interactions are considered in (I), namely Hg1···Cl3 [3.154 (2) Å], Hg2···Cl2 [3.166 (2) Å], Hg2···O1 [2.940 (5) Å] and Hg1···Cl1$^{ii}$ [3.121 (2) Å; symmetry code: (ii) $-x + 1, -y + 2, -z + 2$]. The two last interactions take part in forming a chain along the *a* direction (Fig. 2) and the other two help to stabilize the aggregation with no effect on the pattern of the extended structure formed. Such simultaneously close and distant bonded atoms are usual for mercury(II) (Canty, 1980). A view of the linear chains in the crystal structure of (I) is shown in Fig. 3.

In order to prove the noncovalent nature of the Hg···Cl interaction involving atom Hg1 in the three-coordinate complex, bond-valence-sum calculations were performed using the equation $S_{ij} = \exp[(R_0 - R_{ij})/b_{ij}]$, where $S_{ij}$ is the bond valence portion between atoms *i* and *j*, $R_0$ is the standard value of the bond distance for atoms *i* and *j*, $R_{ij}$ is the actual bond distance between atoms *i* and *j*, and $b_{ij}$ is a constant (Brown & Altermatt, 1985). Such a calculation for the $Hg[Cl]_2[O]$ segment provides a bond-valence-sum (BVS) value of 2.142 valence units (v.u.) for Hg1, considering two Hg—Cl and one Hg—O bonds (with $R_{ij}$ as given in Table 2 for the Hg1—Cl1, Hg1—Cl2 and Hg1—O1 bond lengths, $R_0$ = 2.28 Å for Hg—Cl and 1.972 Å for Hg—O, and $b_{ij}$ = 0.37; Brown, http://www.iucr.org/resources/data/datasets/bond-valence-parameters). Considering the Hg1···Cl1$^{ii}$ and Hg1···Cl3 interactions, the BVSs are calculated as 2.245 and 2.339 v.u., respectively, confirming the noncovalent nature of these interactions.

The P atom in the phosphoric triamide ligand adopts a slightly distorted $P[O][N]_3$ tetrahedral environment and the

**Table 2**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Hg1—Cl1 | 2.301 (2) | P1—O1 | 1.497 (4) |
| Hg1—Cl2 | 2.303 (2) | P1—N1 | 1.625 (6) |
| Hg1—O1 | 2.474 (4) | P1—N3 | 1.634 (6) |
| Hg2—Cl3 | 2.2829 (17) | P1—N2 | 1.636 (6) |
| | | | |
| Cl1—Hg1—Cl2 | 163.79 (8) | P1—O1—Hg1 | 152.6 (3) |
| Cl1—Hg1—O1 | 105.57 (13) | O1—P1—N1 | 109.4 (3) |
| Cl2—Hg1—O1 | 90.58 (12) | O1—P1—N2 | 111.2 (3) |
| Cl3$^{i}$—Hg2—Cl3 | 180.0 | O1—P1—N3 | 114.2 (3) |

Symmetry code: (i) $-x, -y + 2, -z + 2$.

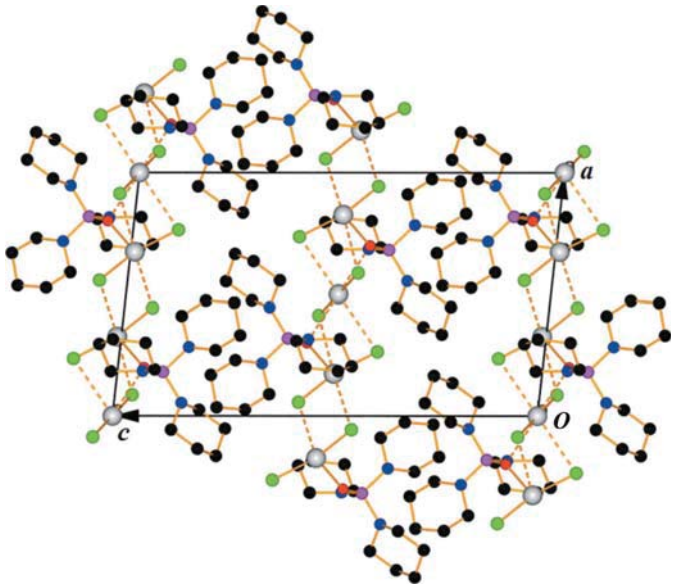



**Figure 3**
A view of the crystal packing of (I), viewed along the *b* axis, showing adjacent linear chains. The Hg···Cl and Hg···O interactions are shown as dashed lines and H atoms have been omitted for clarity.

Hg1—O1—P1 unit is significantly bent, with an angle of 152.6 (3)°. Moreover, the P1═O1 bond length in complex (I) is longer than the P═O double-bond length in phosphoric triamide compounds, for example, longer by about 0.008 Å compared with tris(morpholino)phosphine oxide (BIVYAG; Romming & Songstad, 1982), which is the closest structure to the tris(piperidin-1-yl)phosphane oxide discussed here.

The six-membered piperidine rings in the phosphoric triamide ligand adopt near-chair conformations.

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# supporting information

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## The first phosphoramide–mercury(II) complex with a $Cl_2Hg$–$OP[N(C)(C)]_3$ segment

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### Computing details

Data collection: *X-AREA* (Stoe & Cie, 2009); cell refinement: *X-AREA* (Stoe & Cie, 2009); data reduction: *X-RED32* (Stoe & Cie, 2009); program(s) used to solve structure: SUPERFLIP (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg & Putz, 1999) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

### Dichlorido[tris(piperidin-1-yl)phosphane oxide-$\kappa O$]mercury(II)–dichloridomercury(II) (2/1)

*Crystal data*

$[HgCl_2(C_{15}H_{30}N_3OP)][HgCl_2]$
$M_r$ = 1413.25
Monoclinic, $P2_1/n$
$a$ = 11.1182 (15) Å
$b$ = 10.5995 (8) Å
$c$ = 19.338 (3) Å
$\beta$ = 96.337 (11)°
$V$ = 2265.0 (5) Å$^3$
$Z$ = 2
$F(000)$ = 1340
$D_x$ = 2.072 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 19807 reflections
$\theta$ = 1.1–25.6°
$\mu$ = 10.60 mm$^{-1}$
$T$ = 300 K
Block, less
0.23 × 0.16 × 0.10 mm

*Data collection*

Stoe IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 6.67 pixels mm$^{-1}$
rotation method scans
Absorption correction: integration (*X-SHAPE*; Stoe & Cie, 2002)
$T_{min}$ = 0.066, $T_{max}$ = 0.133
27440 measured reflections
4041 independent reflections
3336 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
$\theta_{max}$ = 25.2°, $\theta_{min}$ = 2.0°
$h$ = −13→13
$k$ = −12→12
$l$ = −23→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.074
$S$ = 1.05
4041 reflections
223 parameters
31 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 5.1731P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.66 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.79 e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. DELU and SIME instructions in *SHELXL97* (Sheldrick, 2008) were applied to C6, C7, C8, C9, and C10 in order to limit the disorder in the corresponding ring. A splitting of the ring in two different parts was not solving the problems of displacement of some atoms in the ring. With these two instructions, the problem is not solved but limited.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.1911 (11) | 0.5083 (7) | 0.9128 (5) | 0.114 (4) |
| H1A | 0.1217 | 0.4599 | 0.9239 | 0.137* |
| H1B | 0.1886 | 0.5125 | 0.8626 | 0.137* |
| C2 | 0.3005 (10) | 0.4461 (10) | 0.9412 (6) | 0.114 (3) |
| H2A | 0.3693 | 0.4895 | 0.9255 | 0.137* |
| H2B | 0.3003 | 0.3604 | 0.9235 | 0.137* |
| C3 | 0.2957 (11) | 0.5713 (9) | 1.0499 (5) | 0.128 (4) |
| H3A | 0.2892 | 0.5622 | 1.0992 | 0.153* |
| H3B | 0.3652 | 0.6243 | 1.0445 | 0.153* |
| C4 | 0.3152 (10) | 0.4423 (9) | 1.0188 (6) | 0.129 (4) |
| H4A | 0.3958 | 0.4126 | 1.0352 | 0.155* |
| H4B | 0.2575 | 0.3831 | 1.0346 | 0.155* |
| C5 | 0.1835 (8) | 0.6340 (8) | 1.0157 (4) | 0.088 (2) |
| H5A | 0.1126 | 0.5889 | 1.0274 | 0.105* |
| H5B | 0.1792 | 0.7198 | 1.0326 | 0.105* |
| C6 | 0.4104 (8) | 0.7807 (10) | 0.8631 (5) | 0.101 (3) |
| H6A | 0.4135 | 0.8382 | 0.9023 | 0.121* |
| H6B | 0.4504 | 0.7031 | 0.8792 | 0.121* |
| C7 | 0.4737 (11) | 0.8352 (14) | 0.8102 (6) | 0.151 (5) |
| H7A | 0.5585 | 0.8434 | 0.8280 | 0.181* |
| H7B | 0.4424 | 0.9194 | 0.8003 | 0.181* |
| C8 | 0.4647 (10) | 0.7608 (12) | 0.7427 (5) | 0.123 (3) |
| H8A | 0.5102 | 0.6828 | 0.7495 | 0.148* |
| H8B | 0.4980 | 0.8096 | 0.7069 | 0.148* |
| C9 | 0.3312 (10) | 0.7319 (12) | 0.7207 (5) | 0.131 (4) |
| H9A | 0.2905 | 0.8094 | 0.7049 | 0.158* |
| H9B | 0.3253 | 0.6739 | 0.6816 | 0.158* |
| C10 | 0.2703 (10) | 0.6786 (12) | 0.7747 (5) | 0.125 (4) |
| H10A | 0.3019 | 0.5947 | 0.7851 | 0.150* |
| H10B | 0.1849 | 0.6705 | 0.7584 | 0.150* |
| C11 | 0.0250 (9) | 0.8724 (8) | 0.7978 (5) | 0.100 (3) |
| H11A | 0.0994 | 0.9109 | 0.7864 | 0.120* |
| H11B | −0.0172 | 0.9336 | 0.8237 | 0.120* |
| C12 | −0.0526 (11) | 0.8387 (12) | 0.7320 (5) | 0.130 (4) |
| H12A | −0.0736 | 0.9149 | 0.7057 | 0.156* |
| H12B | −0.0069 | 0.7847 | 0.7039 | 0.156* |

| | | | | |
|---|---|---|---|---|
| C13 | −0.1650 (10) | 0.7727 (11) | 0.7461 (5) | 0.122 (4) |
| H13A | −0.2095 | 0.7465 | 0.7024 | 0.147* |
| H13B | −0.2159 | 0.8301 | 0.7690 | 0.147* |
| C14 | −0.1355 (9) | 0.6590 (11) | 0.7915 (5) | 0.117 (3) |
| H14A | −0.0926 | 0.5973 | 0.7665 | 0.141* |
| H14B | −0.2098 | 0.6205 | 0.8032 | 0.141* |
| C15 | −0.0569 (8) | 0.6978 (9) | 0.8585 (5) | 0.098 (3) |
| H15A | −0.1020 | 0.7549 | 0.8851 | 0.118* |
| H15B | −0.0361 | 0.6237 | 0.8867 | 0.118* |
| Cl1 | 0.44342 (18) | 1.12472 (19) | 0.93393 (12) | 0.0866 (6) |
| Cl2 | 0.2359 (2) | 0.9726 (2) | 1.10663 (10) | 0.0895 (6) |
| Cl3 | 0.08680 (18) | 1.17631 (17) | 0.95927 (11) | 0.0783 (5) |
| Hg1 | 0.32657 (3) | 1.02803 (3) | 1.00954 (2) | 0.07212 (11) |
| Hg2 | 0.0000 | 1.0000 | 1.0000 | 0.07027 (13) |
| O1 | 0.1987 (4) | 0.8749 (4) | 0.9383 (2) | 0.0708 (12) |
| P1 | 0.18104 (17) | 0.76084 (17) | 0.89264 (9) | 0.0635 (5) |
| N1 | 0.1848 (6) | 0.6350 (5) | 0.9408 (3) | 0.0774 (17) |
| N2 | 0.2835 (5) | 0.7536 (6) | 0.8382 (3) | 0.0714 (15) |
| N3 | 0.0539 (5) | 0.7600 (5) | 0.8414 (3) | 0.0689 (15) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.192 (12) | 0.061 (5) | 0.084 (6) | 0.014 (6) | −0.010 (7) | −0.005 (4) |
| C2 | 0.123 (8) | 0.091 (6) | 0.128 (9) | 0.028 (6) | 0.009 (7) | 0.006 (6) |
| C3 | 0.180 (11) | 0.096 (7) | 0.089 (6) | −0.010 (7) | −0.060 (7) | 0.012 (5) |
| C4 | 0.127 (8) | 0.077 (6) | 0.165 (11) | 0.016 (6) | −0.065 (7) | 0.016 (6) |
| C5 | 0.109 (7) | 0.075 (5) | 0.082 (6) | 0.008 (5) | 0.019 (5) | 0.002 (4) |
| C6 | 0.086 (6) | 0.127 (8) | 0.092 (6) | −0.005 (5) | 0.017 (5) | −0.036 (5) |
| C7 | 0.122 (8) | 0.221 (13) | 0.114 (8) | −0.076 (8) | 0.033 (6) | −0.047 (7) |
| C8 | 0.113 (6) | 0.164 (10) | 0.099 (6) | −0.024 (7) | 0.039 (6) | −0.021 (6) |
| C9 | 0.133 (8) | 0.192 (11) | 0.070 (6) | −0.052 (8) | 0.014 (5) | −0.012 (6) |
| C10 | 0.115 (7) | 0.178 (10) | 0.086 (6) | −0.032 (7) | 0.030 (6) | −0.043 (6) |
| C11 | 0.106 (7) | 0.087 (6) | 0.100 (7) | 0.003 (5) | −0.023 (5) | 0.022 (5) |
| C12 | 0.137 (10) | 0.147 (10) | 0.099 (8) | 0.007 (8) | −0.020 (7) | 0.014 (7) |
| C13 | 0.110 (9) | 0.141 (10) | 0.105 (8) | 0.020 (7) | −0.037 (6) | −0.013 (7) |
| C14 | 0.105 (8) | 0.137 (9) | 0.103 (7) | −0.031 (7) | −0.021 (6) | −0.015 (6) |
| C15 | 0.085 (6) | 0.115 (7) | 0.091 (6) | −0.017 (5) | −0.003 (5) | 0.010 (5) |
| Cl1 | 0.0726 (12) | 0.0741 (12) | 0.1148 (16) | 0.0062 (10) | 0.0178 (11) | 0.0170 (11) |
| Cl2 | 0.0850 (13) | 0.1108 (16) | 0.0726 (12) | 0.0067 (12) | 0.0082 (10) | −0.0037 (11) |
| Cl3 | 0.0775 (12) | 0.0641 (10) | 0.0962 (14) | −0.0030 (9) | 0.0231 (10) | 0.0111 (9) |
| Hg1 | 0.06359 (18) | 0.06830 (18) | 0.0848 (2) | 0.00149 (14) | 0.00990 (14) | −0.00687 (14) |
| Hg2 | 0.0711 (3) | 0.0643 (2) | 0.0750 (3) | −0.01132 (18) | 0.0064 (2) | 0.00707 (18) |
| O1 | 0.083 (3) | 0.058 (3) | 0.071 (3) | −0.006 (2) | 0.006 (2) | −0.014 (2) |
| P1 | 0.0709 (12) | 0.0579 (10) | 0.0600 (11) | 0.0033 (9) | −0.0001 (9) | −0.0061 (8) |
| N1 | 0.113 (5) | 0.052 (3) | 0.063 (4) | 0.013 (3) | −0.008 (3) | −0.002 (3) |
| N2 | 0.069 (4) | 0.077 (4) | 0.067 (4) | −0.004 (3) | 0.004 (3) | −0.016 (3) |
| N3 | 0.065 (4) | 0.068 (4) | 0.071 (4) | −0.001 (3) | −0.001 (3) | 0.006 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.438 (13) | C10—N2 | 1.457 (10) |
| C1—N1 | 1.452 (9) | C10—H10A | 0.9700 |
| C1—H1A | 0.9700 | C10—H10B | 0.9700 |
| C1—H1B | 0.9700 | C11—N3 | 1.475 (9) |
| C2—C4 | 1.493 (13) | C11—C12 | 1.500 (12) |
| C2—H2A | 0.9700 | C11—H11A | 0.9700 |
| C2—H2B | 0.9700 | C11—H11B | 0.9700 |
| C3—C5 | 1.501 (12) | C12—C13 | 1.483 (15) |
| C3—C4 | 1.518 (14) | C12—H12A | 0.9700 |
| C3—H3A | 0.9700 | C12—H12B | 0.9700 |
| C3—H3B | 0.9700 | C13—C14 | 1.507 (14) |
| C4—H4A | 0.9700 | C13—H13A | 0.9700 |
| C4—H4B | 0.9700 | C13—H13B | 0.9700 |
| C5—N1 | 1.450 (9) | C14—C15 | 1.536 (11) |
| C5—H5A | 0.9700 | C14—H14A | 0.9700 |
| C5—H5B | 0.9700 | C14—H14B | 0.9700 |
| C6—C7 | 1.426 (13) | C15—N3 | 1.467 (10) |
| C6—N2 | 1.469 (10) | C15—H15A | 0.9700 |
| C6—H6A | 0.9700 | C15—H15B | 0.9700 |
| C6—H6B | 0.9700 | Hg1—Cl1 | 2.301 (2) |
| C7—C8 | 1.519 (13) | Hg1—Cl2 | 2.303 (2) |
| C7—H7A | 0.9700 | Hg1—O1 | 2.474 (4) |
| C7—H7B | 0.9700 | Hg2—Cl3 | 2.2829 (17) |
| C8—C9 | 1.530 (14) | Hg2—Cl3$^{i}$ | 2.2829 (18) |
| C8—H8A | 0.9700 | P1—O1 | 1.497 (4) |
| C8—H8B | 0.9700 | P1—N1 | 1.625 (6) |
| C9—C10 | 1.423 (13) | P1—N3 | 1.634 (6) |
| C9—H9A | 0.9700 | P1—N2 | 1.636 (6) |
| C9—H9B | 0.9700 | | |

| | | | |
|---|---|---|---|
| C2—C1—N1 | 110.8 (8) | C9—C10—H10A | 109.0 |
| C2—C1—H1A | 109.5 | N2—C10—H10A | 109.0 |
| N1—C1—H1A | 109.5 | C9—C10—H10B | 109.0 |
| C2—C1—H1B | 109.5 | N2—C10—H10B | 109.0 |
| N1—C1—H1B | 109.5 | H10A—C10—H10B | 107.8 |
| H1A—C1—H1B | 108.1 | N3—C11—C12 | 111.2 (8) |
| C1—C2—C4 | 113.0 (9) | N3—C11—H11A | 109.4 |
| C1—C2—H2A | 109.0 | C12—C11—H11A | 109.4 |
| C4—C2—H2A | 109.0 | N3—C11—H11B | 109.4 |
| C1—C2—H2B | 109.0 | C12—C11—H11B | 109.4 |
| C4—C2—H2B | 109.0 | H11A—C11—H11B | 108.0 |
| H2A—C2—H2B | 107.8 | C13—C12—C11 | 112.0 (9) |
| C5—C3—C4 | 111.9 (7) | C13—C12—H12A | 109.2 |
| C5—C3—H3A | 109.2 | C11—C12—H12A | 109.2 |
| C4—C3—H3A | 109.2 | C13—C12—H12B | 109.2 |
| C5—C3—H3B | 109.2 | C11—C12—H12B | 109.2 |

| | | | |
|---|---|---|---|
| C4—C3—H3B | 109.2 | H12A—C12—H12B | 107.9 |
| H3A—C3—H3B | 107.9 | C12—C13—C14 | 110.5 (9) |
| C2—C4—C3 | 111.6 (8) | C12—C13—H13A | 109.6 |
| C2—C4—H4A | 109.3 | C14—C13—H13A | 109.6 |
| C3—C4—H4A | 109.3 | C12—C13—H13B | 109.6 |
| C2—C4—H4B | 109.3 | C14—C13—H13B | 109.6 |
| C3—C4—H4B | 109.3 | H13A—C13—H13B | 108.1 |
| H4A—C4—H4B | 108.0 | C13—C14—C15 | 110.1 (9) |
| N1—C5—C3 | 110.1 (7) | C13—C14—H14A | 109.6 |
| N1—C5—H5A | 109.6 | C15—C14—H14A | 109.6 |
| C3—C5—H5A | 109.6 | C13—C14—H14B | 109.6 |
| N1—C5—H5B | 109.6 | C15—C14—H14B | 109.6 |
| C3—C5—H5B | 109.6 | H14A—C14—H14B | 108.1 |
| H5A—C5—H5B | 108.2 | N3—C15—C14 | 110.2 (7) |
| C7—C6—N2 | 112.1 (8) | N3—C15—H15A | 109.6 |
| C7—C6—H6A | 109.2 | C14—C15—H15A | 109.6 |
| N2—C6—H6A | 109.2 | N3—C15—H15B | 109.6 |
| C7—C6—H6B | 109.2 | C14—C15—H15B | 109.6 |
| N2—C6—H6B | 109.2 | H15A—C15—H15B | 108.1 |
| H6A—C6—H6B | 107.9 | Cl1—Hg1—Cl2 | 163.79 (8) |
| C6—C7—C8 | 114.5 (10) | Cl1—Hg1—O1 | 105.57 (13) |
| C6—C7—H7A | 108.6 | Cl2—Hg1—O1 | 90.58 (12) |
| C8—C7—H7A | 108.6 | Cl3$^{i}$—Hg2—Cl3 | 180.0 |
| C6—C7—H7B | 108.6 | P1—O1—Hg1 | 152.6 (3) |
| C8—C7—H7B | 108.6 | O1—P1—N1 | 109.4 (3) |
| H7A—C7—H7B | 107.6 | N1—P1—N3 | 108.0 (3) |
| C7—C8—C9 | 108.2 (9) | O1—P1—N2 | 111.2 (3) |
| C7—C8—H8A | 110.0 | O1—P1—N3 | 114.2 (3) |
| C9—C8—H8A | 110.0 | N1—P1—N2 | 110.8 (3) |
| C7—C8—H8B | 110.0 | N3—P1—N2 | 103.1 (3) |
| C9—C8—H8B | 110.0 | C5—N1—C1 | 111.8 (6) |
| H8A—C8—H8B | 108.4 | C5—N1—P1 | 125.2 (5) |
| C10—C9—C8 | 113.6 (9) | C1—N1—P1 | 123.0 (5) |
| C10—C9—H9A | 108.8 | C10—N2—C6 | 112.8 (7) |
| C8—C9—H9A | 108.8 | C10—N2—P1 | 123.7 (6) |
| C10—C9—H9B | 108.8 | C6—N2—P1 | 119.5 (5) |
| C8—C9—H9B | 108.8 | C15—N3—C11 | 110.8 (6) |
| H9A—C9—H9B | 107.7 | C15—N3—P1 | 123.9 (5) |
| C9—C10—N2 | 113.0 (9) | C11—N3—P1 | 117.5 (5) |

| | | | |
|---|---|---|---|
| N1—C1—C2—C4 | 55.8 (13) | N3—P1—N1—C1 | 66.0 (9) |
| C1—C2—C4—C3 | −49.4 (14) | N2—P1—N1—C1 | −46.3 (9) |
| C5—C3—C4—C2 | 47.2 (14) | C9—C10—N2—C6 | −53.4 (13) |
| C4—C3—C5—N1 | −52.2 (11) | C9—C10—N2—P1 | 149.3 (8) |
| N2—C6—C7—C8 | −52.8 (14) | C7—C6—N2—C10 | 52.7 (12) |
| C6—C7—C8—C9 | 50.3 (15) | C7—C6—N2—P1 | −149.0 (8) |
| C7—C8—C9—C10 | −50.1 (15) | O1—P1—N2—C10 | −160.6 (8) |
| C8—C9—C10—N2 | 53.2 (15) | N1—P1—N2—C10 | 77.5 (8) |

| | | | |
|---|---|---|---|
| N3—C11—C12—C13 | 55.9 (12) | N3—P1—N2—C10 | −37.8 (8) |
| C11—C12—C13—C14 | −54.6 (13) | O1—P1—N2—C6 | 43.6 (7) |
| C12—C13—C14—C15 | 55.0 (13) | N1—P1—N2—C6 | −78.3 (7) |
| C13—C14—C15—N3 | −57.4 (12) | N3—P1—N2—C6 | 166.4 (6) |
| Hg1—O1—P1—N1 | 83.1 (7) | C14—C15—N3—C11 | 58.7 (10) |
| Hg1—O1—P1—N3 | −155.7 (6) | C14—C15—N3—P1 | −152.9 (7) |
| Hg1—O1—P1—N2 | −39.5 (7) | C12—C11—N3—C15 | −57.9 (11) |
| C3—C5—N1—C1 | 59.3 (10) | C12—C11—N3—P1 | 151.5 (7) |
| C3—C5—N1—P1 | −120.2 (7) | O1—P1—N3—C15 | −94.5 (7) |
| C2—C1—N1—C5 | −61.4 (11) | N1—P1—N3—C15 | 27.4 (7) |
| C2—C1—N1—P1 | 118.1 (8) | N2—P1—N3—C15 | 144.6 (7) |
| O1—P1—N1—C5 | 10.2 (8) | O1—P1—N3—C11 | 51.9 (7) |
| N3—P1—N1—C5 | −114.6 (7) | N1—P1—N3—C11 | 173.8 (6) |
| N2—P1—N1—C5 | 133.1 (7) | N2—P1—N3—C11 | −68.9 (7) |
| O1—P1—N1—C1 | −169.2 (8) | | |

Symmetry code: (i) −*x*, −*y*+2, −*z*+2.